Cryogenic Modules for Synergistic O_2 Generation and CO_2 Retention in Closed-Circuit Escape Respirators

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Abstract. Since 2018, NASA and the National Institute for Occupational Safety and Health (NIOSH) have been developing a liquid oxygen storage module (LOXSM) based on the NASA patent-pending Cryogenic Flux Capacitor (CFC) technology. LOXSM's could potentially replace the gaseous or chemical-based oxygen supply in current closed-circuit escape respirators (CCER), with reducing CCER size being a primary goal. By virtue of the CFC functionality, cryogenic oxygen stored within the LOXSM is released in response to heat input, ideally from the breathing loop. Prior efforts focused on the oxygen storage potential of silica aerogel materials that the CFC utilizes, and were previously reported. Current work explored the LOXSM's potential to remove and retain CO_2 produced by the CCER user, in conjunction with oxygen generation, creating a synergy that may be exploited to reduce or eliminate the chemical CO_2 absorber used in current CCERs. A test program for determining CO_2 retention is presented, as well as the evolution of LOXSM prototypes. Testing showed that it is possible to completely remove CO_2 out of an effluent stream at the flowrate required for the capacity of the CCER for an appreciable time, and that the LOXSM prototype design progression had a positive effect on that duration.

1. Introduction

Closed-circuit escape respirators (CCERs) are used for escape in an emergency, such as an explosion in an underground coal mine, which makes the ambient air inside the mine irrespirable. CCERs need to be small-sized and as light as possible as they must be carried on the person, ready to be quickly deployed and donned immediately when the emergency happens, and to have an independent source of oxygen isolated from the ambient atmosphere. In the design of self-contained breathing apparatus, using the closed-circuit principle where the breathing gas from the user is recirculated and reconditioned in a closed loop within the apparatus is the most efficient and allows for a device in a small size. CCERs operate on this closed-circuit principle where the user breathes the same gas volume in a closed loop. The breathing gas is conditioned in the respirator to maintain the required oxygen (O_2) content and remove the generated carbon dioxide (CO_2). Oxygen is added to the breathing loop in the CCER, and carbon dioxide exhaled by the user is absorbed by a scrubber material that bonds it through a chemical reaction. The O_2 and CO_2 levels in the CCER are maintained according to the user's metabolic needs at varying workloads. In current CCER designs, the oxygen addition may either be from a high-pressure gas cylinder-based oxygen delivery system (ODS) or an oxygen-generating chemical, such as potassium

superoxide (KO_2). A CCER design must be approved under the applicable provisions of Title 42 CFR Part 84 sub-part O for use in the United States. Further, the design must meet MSHA regulations of Title 30 CFR 75.1714-1 for use in US underground coal mines.

2. LOXSM Design Approach

The liquid oxygen storage module (LOXSM) is an innovative concept to store oxygen in solid-state form, according to physisorption processes at any cryogenic temperature, and deliver it as a gas using the cryogenic flux capacitor[1,2] (CFC) technology as the core storage element. CFCs employ nano-porous aerogel composites to store large quantities of fluid molecules in a physisorbed state at moderate pressures and cryogenic temperatures. The LOXSM will be designed to be integrated into a CCER and charged with liquid oxygen (LOX) prior to use. Gaseous oxygen would be admitted into the breathing loop of the CCER by introducing heat into the LOXSM. The source of this heat will be a combination of environmental (i.e. heat leak into the system) and from the user's effluent breathing stream during use. Further, the CFC technology could potentially reduce the size of the necessary carbon dioxide absorber used in current CCERs by sequestering CO_2 from the exhaled air stream, or this technology could potentially even eliminate the need for the CO_2 absorber altogether if 100% of this gas can be removed.

Through prior work, various aerogel materials have been characterized for their storage performance, and small-scale prototype LOXSMs have been designed and tested[3]. Recently, focus has been on devising LOXSMs aimed at CO_2 sequestration in addition to O_2 generation. These designs must be capable of providing oxygen into the breathing loop of the CCER according to the user's metabolic consumption, while retaining all or some of the CO_2 produced by the user. If successfully developed, such a LOXSM could replace the traditional ODS and supplement or eliminate the bulky CO_2 scrubber material in the CCER.

All LOXSM designs must comply with the CAP 3 requirements in Title 42 CFR Part 84 sub-part O for 81 liters oxygen capacity as given in 42CFR84.304 Table 2. The applicable parameters are as follows:

- Volume of oxygen consumed per minute (i.e., the required O_2 generation rate), $VO_2 = 1.35$ L/min
- Volume of carbon dioxide produced per minute (i.e., the target CO_2 sequestration rate), $VCO_2 = 1.15$ L/min
- Average inhaled $O_2 > 19.5\%$, average inhaled $CO_2 < 1.5\%$ with acceptable excursion ranges as given in 42CFR84.304-Table 1

With a nominal use time of 1 hour at VO_2 and VCO_2, the LOXSM must provide a minimum of 81 L of oxygen during CCER use, which translates to an equivalent LOX volume of roughly 100 mL, and sequester 69 L of CO_2. Using Cryogel® aerogel blanket material from Aspen Aerogels as the baseline LOXSM storage media, which has a LOX volume ratio[2] of 1.03:1, the minimum required aerogel volume to store 100 mL of LOX is 97 mL. Additional material must be included, however, to account for the dormancy time required prior to CCER use, as environmental heat leak into the LOXSM will slowly deplete the unit. For the current work, designing for dormancy time was not the key focus—hence this is left to future work.

A guiding principle for the design of LOXSMs aimed at CO_2 removal was maximizing the "cold path" that the incoming exhaled breathing stream must take through the unit. As the exhaled stream enters the unit, it must follow a pre-determined flow path per the design. In a LOXSM, this flow path can double as the CFC, resulting in the warm, CO_2 rich effluent stream coming into direct contact with the cryogenic aerogel at 90 K, well below the freezing point of CO_2 at 216.6 K. The cold path is the physical and temporal length the gas is in contact with the cold CFC, and maximizing it leads to a higher probability that the CO_2 will be trapped within the LOXSM (sequestered) and removed from the breathing loop. This process imparts heat to the aerogel, which discharges oxygen into the loop in response, leading to a sort of synergy between the two processes. An additional benefit may be in

desiccating the moist breathing stream, as most/all of the water vapor would likely be frozen out within the LOXSM. However, moisture testing was not within the scope of the current work—hence this remains an additional topic of future study.

3. Evolution of LOXSM for CO_2 Sequestration

A total of four prototypes were designed, each guided by the requirements and approach laid out above, and building off the knowledge gained through testing. Testing consisted of using either pure (99.8%) CO_2 from a high-pressure gas cylinder or a predetermined mixture of gases that mimicked the composition of the exhaled breath (5% CO_2, 16% O_2, and 79% N_2). After charging the prototype with liquid nitrogen (LN_2) and configuring the test setup, the source gas was passed through it at a constant flow rate dictated by the particular test, and the exit stream was analyzed using a INFICON brand Transpector® CPM residual gas analyzer (RGA) to determine the compositional breakdown.

3.1 Prototype Version 1.0

An initial small CFC was fabricated and tested using LN_2 to determine the feasibility of the device to sequester CO_2. This prototype, deemed version 1.0 (v1.0), consisted of a 5-mm-thick Cryogel® blanket spirally wound onto a 9.5-mm-diameter G-10 fiberglass epoxy tube to create a cylindrical geometry 50.8 mm in diameter by 76.2 mm long. The G-10 tube had radial holes drilled along its length to direct gas into the middle of the CFC, and this tube could act as either a feed or discharge depending on the direction of flow. The estimated aerogel mass for prototype v1.0 was 24-g, which translated to roughly 155-mL of LN_2 when saturated, or 149-mL of LOX.

Prototype v1.0 was configured into a custom-designed, 3-D printed cap, that interfaced to a commercially available vacuum-insulated vessel for testing. The cap had three 9.5-mm-diameter ports to accommodate various flow configurations. The center port was used to interface the cap to the CFC via the G-10 feed tube; one was used to pass a 9.5-mm-diameter flexible tube through the cap and position its outlet at the bottom of the vessel; and the last port was left open to the gas space at the top of the vessel. Figure 1 shows prototype v1.0 disassembled and during initial benchtop testing.

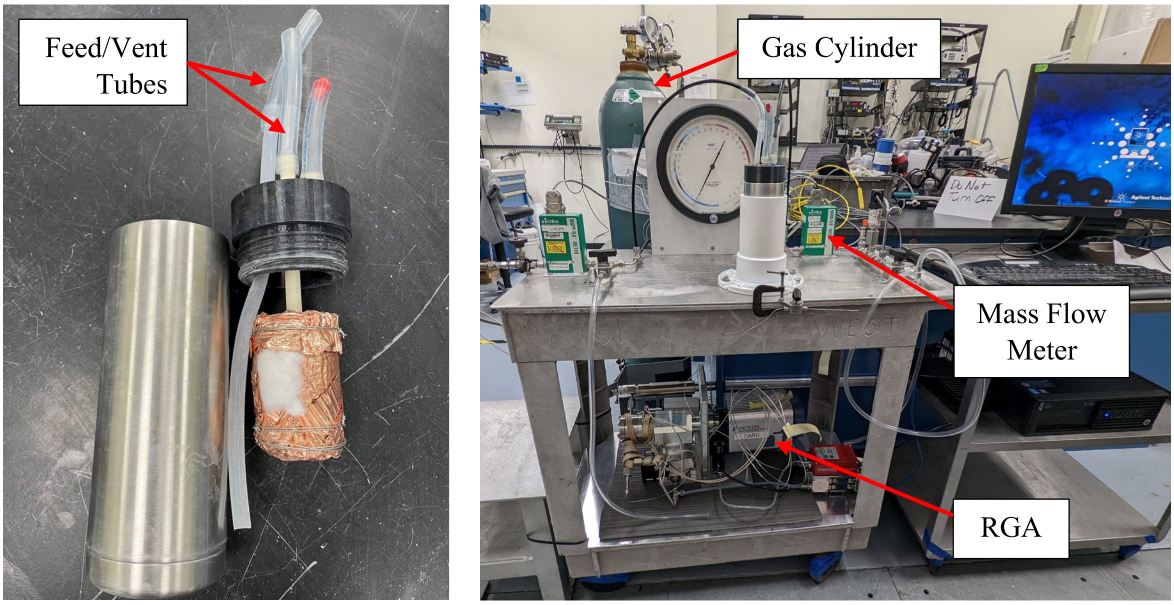

Figure 1. Prototype v1.0 configuration (left) and during testing (right)

Prototype v1.0 was tested extensively and yielded encouraging results for the utility of a CFC to successfully sequester CO_2 from a breathing stream. Figure 2 presents the results from a single test using the simulated exhaled breath source gas at a constant flow rate of 1600-sccm (not compensated for the gas mixture), with the gas introduced into the middle of the CFC via the feed tube.

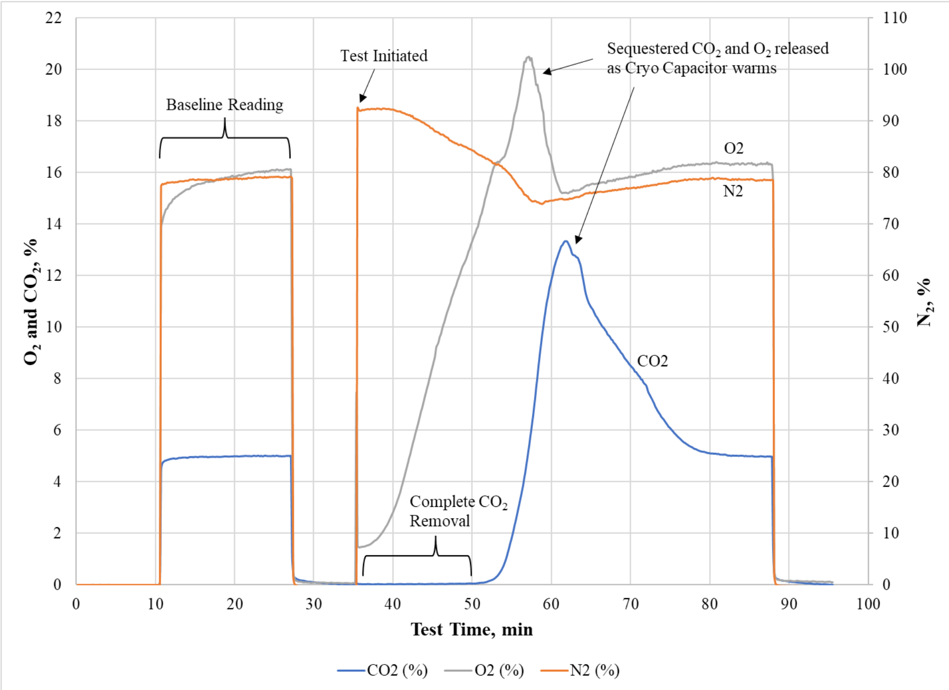

Figure 2. Prototype v1.0 test results: exhaled breath gas mixture, 1600 sccm flow rate, gas fed into the middle of the CFC

Inspection of Figure 2 shows the different test phases, with the baseline reading being measured by bypassing the prototype altogether and analyzing the composition of the gas cylinder directly using the RGA. Testing was then initiated by rerouting the gas though the prototype and was marked by the nitrogen percentage spiking to over 90% while the CO_2 percentage was zero. Oxygen percentage also decreased; however, in an actual LOXSM this would not be the case as the unit would be charged with LOX instead of LN_2. In total, the operational time with complete CO_2 removal was roughly 15 minutes, which translated to an estimated CO_2 volume and mass of 1.35-L and 2.24-g, respectively.

3.2 Prototype Version 2.0

Drawing on the positive test results from the small prototype v1.0, version 2.0 was specifically designed to be both physically larger, and hence to possess a larger storage capacity, and to have a focused, long cold path through the unit. For packaging efficiency, a flat, round geometry was chosen that employed a spiral flow path, with a gas outlet port sized to accommodate a 12.5-mm-diameter tube located in the center of the base plate and an inlet port sized for a 9.5-mm-diameter tube on the curved wall, oriented roughly tangentially. Approximate overall dimensions of prototype v2.0 were (not including the gas supply tubes), 127-mm in diameter by 41.4-mm tall. The spiral flow path measured 35.3-mm tall with a width of 13-mm—this volume would house the CFC, consisting of aerogel blanket strips interspersed with corrugated mesh foils to allow for quick LN_2 charging of the unit and to provide an adequate flow area to decrease the pressure drop through the prototype while in use.

Four strips of 5-mm-thick Cryogel® and two corrugated foils (1.6-mm peak-to-peak) made up the CFC—i.e., two flat, spiral Cryogel® strips, bookending two vertical strips, separated by two mesh foils. The total aerogel blanket mass for prototype v2.0 was 35-g, translating to roughly 225.8-mL and 218-mL of stored LN_2 and LOX, respectively.

Due to the complexity of the design, the unit was 3-D printed from Ultem® material—a high-performance engineering plastic that, in 3-D printed form, has been successfully used for various cryogenics applications in the past at the Cryogenics Test Laboratory (CTL) at NASA Kennedy Space Center (KSC). Figure 3 presents graphics of the prototype, calling out relevant features, as well as the final 3-D printed body prior to assembly.

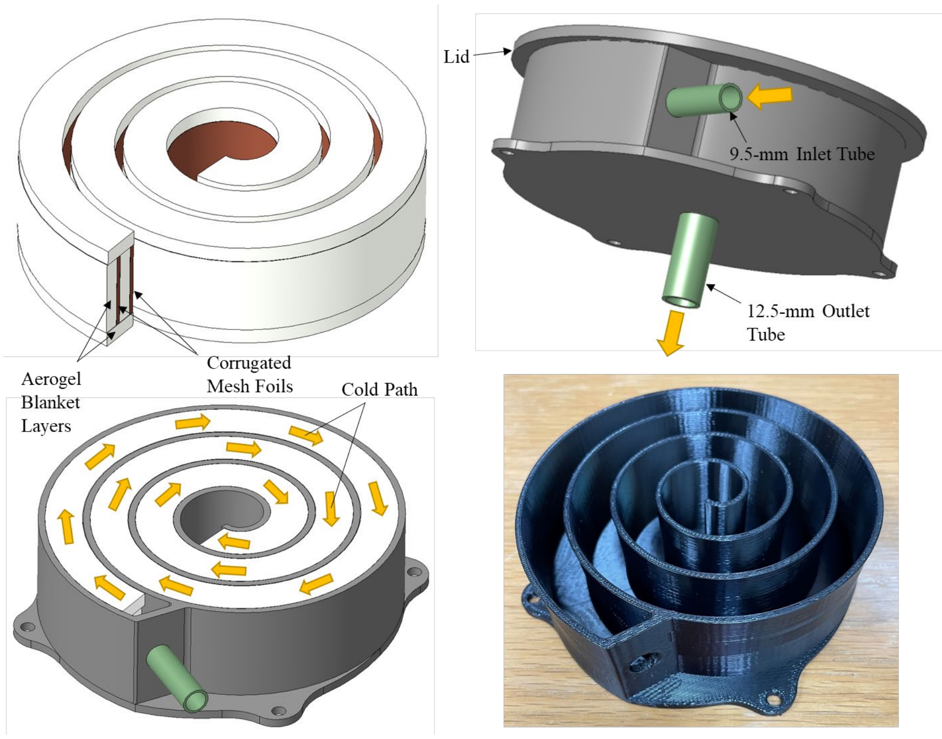

Figure 3. Prototype v2.0: CAD renderings of CFC (upper left); gas inlet/outlet layout (upper right), and spiral flow path (lid removed, lower left); and final 3-D printed body (lower right)

For testing, prototype v2.0 was configured similarly to v1.0, only using a larger, vacuum-insulated container—a Stanley brand Adventure Series 2.8-L vacuum crock. The outlet of the prototype was sealed to the lid of the vacuum container, with the inlet open to the inner volume. Gas was fed through the lid of the container, slightly pressurizing the inner volume and forcing flow through the prototype. The prototype was saturated with LN_2 outside the container and then interfaced to the crock to begin testing. Figure 4 shows the chilled prototype on the lid prior to testing and the final test configuration.

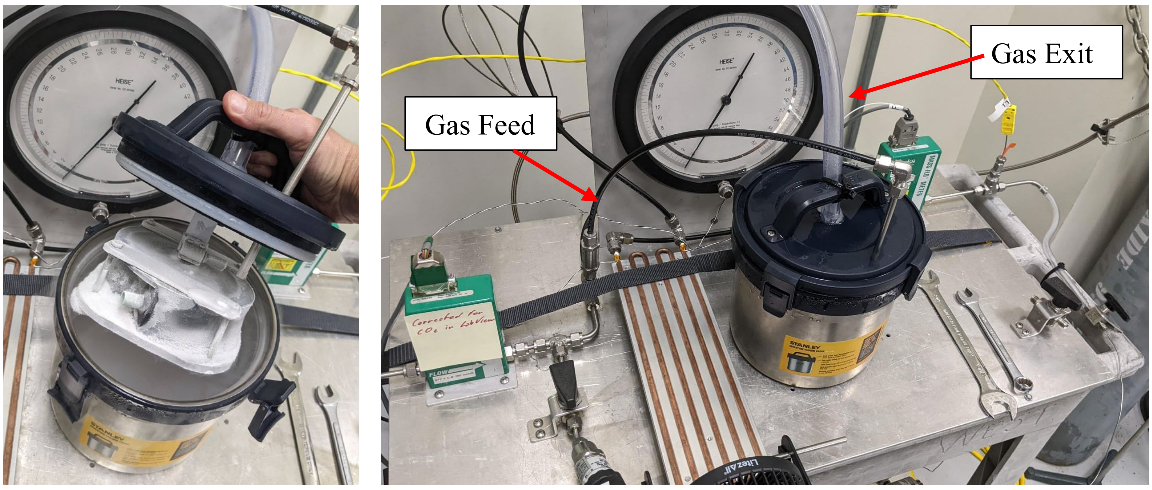

Figure 4. Prototype v2.0 chilled using LN_2 prior to testing (left); final test configuration (right)

Prototype v2.0 was also tested extensively, using both 99.8% CO_2 as well as the gas mixture, yielding excellent results. Figure 5 presents results using the gas mixture at a flow rate of 1599-sccm (not compensated for the gas mixture) and closely matches those seen in v1.0 testing in Figure 2, with the most significant difference being the extended duration for complete CO_2 sequestration. In total, the

operational time with complete CO_2 removal from the test presented in Figure 5 was roughly 80 minutes—over 5 times longer than prototype v1.0—which translated to an estimated CO_2 volume and mass of 6.96-L and 13.8-g, respectively.

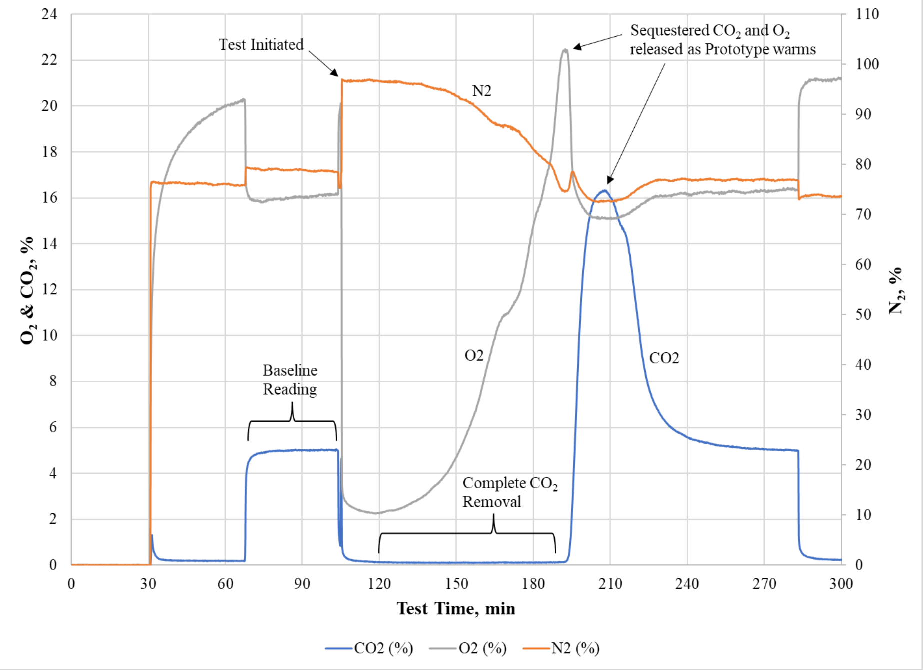

Figure 5. Prototype v2.0 Test Results: Exhaled Breath Gas Mixture, 1599-sccm Flow Rate

Testing of prototype v2.0 with 99.8% CO_2 produced similar results as the gas mixture, although with a decreased capture time but an increased total sequestration. Over three tests, at a supply rate of 1160-sccm (compensated for pure CO_2), the average sequestration time dropped to 34.5 min, but the total mass and volume captured rose to 78.7-g and 39.8-L, respectively. This performance, as intended, matched the target CO_2 capture rate of 1.15 L/min from Section 2 almost exactly, conforming the potential for a LOXSM to capture an adequate quantity of CO_2. However, comparison to the gas mixture performance reveals that designing for both proper CO_2 sequestration quantity and duration could be difficult. Ultimately, the heat input into the unit during operation dictates the duration, and the capturing of CO_2 constitutes only a single mode of the total heat absorption. As such, the evolution from prototype v2.0 to v3.0 focused once again on a physical scale-up, with the testing goal to demonstrate the unit using LOX instead of LN_2, and on a standard breathing apparatus that more closely duplicates the true use scenario, including flow rates, gas composition, and heat loads.

3.3 Prototype Version 3.0 & 3.1

The largest design departure from prototype v2.0 to v3.0 was in overall geometry. Moving away from the flat cylindrical shape and spiral cold path to a prismatic design with a serpentine cold path, v3.0 allowed for more simplified fluid interfaces that were not required to be perpendicular to one another, and also simplified construction of the CFC using precision-cut, flat sheets of aerogel blanket and corrugated foils instead of rounds strips. Capitalizing on the success in 3-D printing v2.0 out of Ultem® material, additional advanced features were also included in the v3 designs that aimed to combat the aforementioned dormancy issue by utilizing vapor shielding to cut down on environmental heat leak. These features—essentially consisting of built-in flow channels in five of the six walls of the unit—increased the overall complexity of the prototype, resulting in a three-piece construction, 3-D printed from Ultem®, that required assembly using cryogenic epoxy (Stycast 2850FT with 23 LV catalyst) to seal the different flow paths and cavities.

Designs for prototypes v3.0 and v3.1 were very similar, with the latter expanded to accommodate around twice the aerogel volume, but only v3.1 was printed and assembled. Overall dimensions for v3.0 were 124-mm long x 115.6-mm tall x 57-mm deep, with 9.5-mm dimeter 12.5-mm diameter gas inlet and outlet ports, respectively; with v3.1 expanded to 226-mm long x 124.5-mm tall x 59-mm deep, with equal-sized, 9.5-mm-diameter ports (due to printing constraints). Estimated aerogel mass for v3.0 was 62-g, translating to 406-mL and 391-mL of stored LN_2 and LOX, respectively, but was expanded to 130-g for prototype v3.1, yielding estimates for the LN_2 and LOX storage capacities of 840-mL and 810-mL respectively. Figures 6 through 8 present the design for the v3.0 prototype, and v3.1 during assembly.

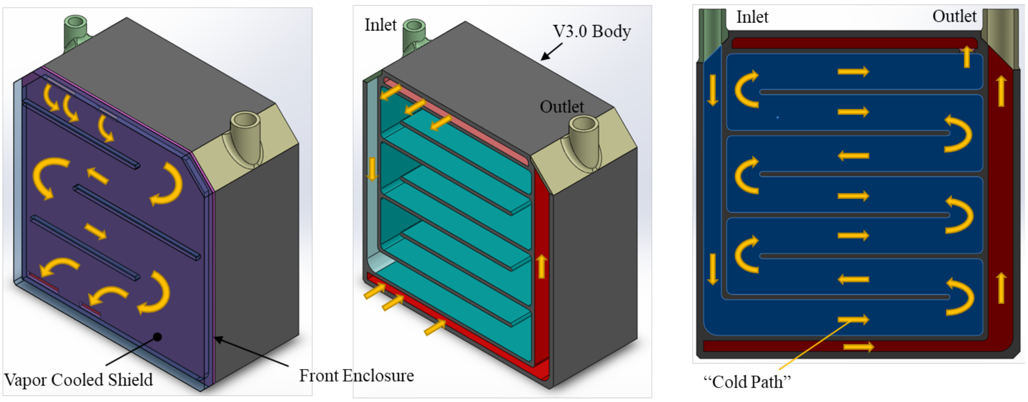

Figure 6. CAD views of prototype v3.0 showing flow/cold paths: full assembly (left); body only (middle and right)

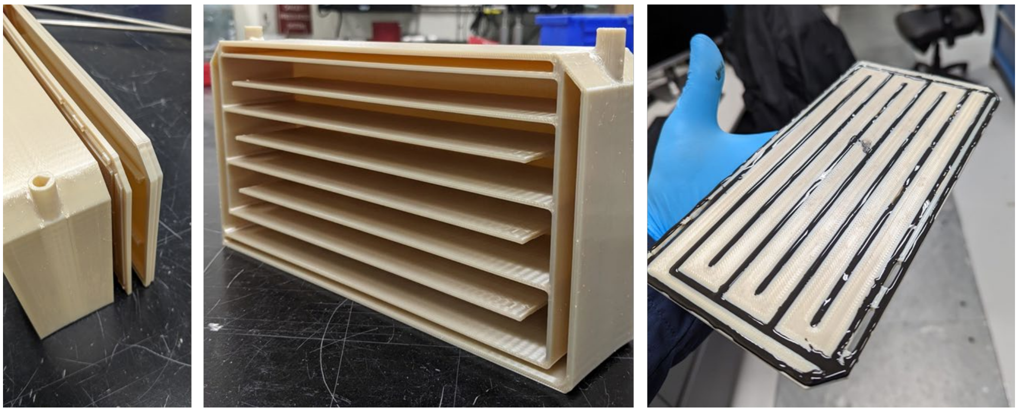

Figure 7. 3-D printed prototype v3.1 parts prior to assembly (left and middle); front enclosure with Stycast 2850FT epoxy (black) during final assembly (right)

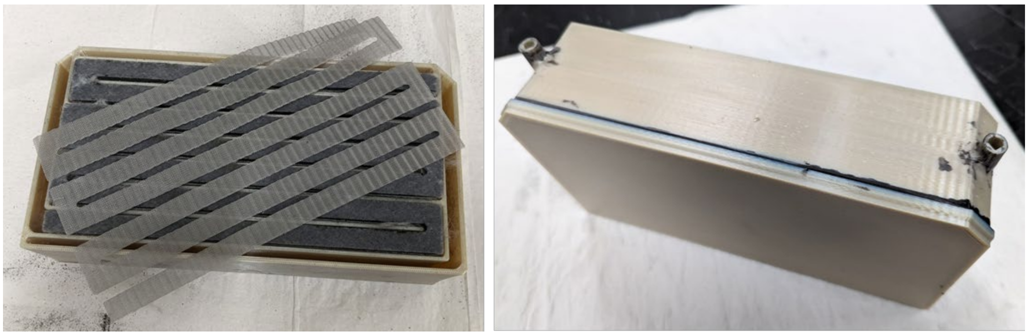

Figure 8. Prototype v3.1 with CFC installed—stack-up of nine 5-mm-thick Spaceloft Subsea® aerogel blanket layers with corrugated aluminum mesh foils (left); and final v3.1 assembly (right)

As can be seen from Figure 8, prototype v3.1 has been fully assembled and is currently awaiting CO_2 sequestration and dormancy time testing using LN_2 at the Cryogenics Test Laboratory, with plans for more rigorous, operational testing using LOX on the integrated component test platform (ICTP) at NIOSH NPPTL at a later stage.

4. Future Work

Although the test data and evolution in design of LOXSM prototypes presented in this work lends itself to a potential synergy existing within the technology for simultaneous oxygen supply and CO_2 sequestration for use in CCERs, much more maturation work is necessary. Of particular importance, and a primary goal for the planned continuation of the current work, is testing in LOX instead of LN_2, as the performance, although expected to be roughly equivalent for most metrics, will undoubtedly change. Also, designing for proper dormancy time remains a top technical issue for real-world implementation into CCER applications and will require further attention, including high-performance insulation system design for the LOXSM and exploration of advanced heat management approaches such as vapor shielding, implemented but as-yet untested in prototype v3.1.

5. Conclusion

Continued development of the Liquid Oxygen Storage Module (LOXSM) technology for Closed-Circuit Escape Respirators (CCER) was presented and discussed, focusing on exploiting the potential synergy between the required oxygen generation/supply to the CCER and CO_2 capture from the exhaled breath though cryogenic freezing/sequestration.

An evolution of prototypes was presented that employed the "cold path" approach to maximize the quantity and time of CO_2 capture, and this approach was experimentally verified using both 99.8% CO_2 as well as a mixture of gases that mimicked the composition of exhaled breath (5% CO_2, 16% O_2, and 79% N_2). Testing also confirmed that complete CO_2 capture is possible in appreciable quantities and over long durations while simultaneously releasing stored cryogenic nitrogen (the surrogate for liquid oxygen used during testing). Results are promising for the use of LOXSMs to potentially reduce the mass and/or volume of current CCERs by eliminating the need for chemical CO_2 scrubber materials.

6. Disclaimer

The findings and conclusions in this report are those of the authors and do not necessarily represent the official position of the National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention. Mention of any company or product does not constitute endorsement by the National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention.

7. References

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